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SOLID LUBRICANTS FOR HIGH TEMPERATURE METALWORKING

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INTRODUCTION

Metalworking involving severe plastic deformation, particularly at high temperatures, can be strongly influenced by friction and lubrication effects (refs. 1 to 4 and 7). Different lubricants (e.g., oils, soaps, paraffin, graphite, MoS_2 and glass) have been used with varying degrees of success. At extreme temperatures, glass has been used successfully in the extrusion process (ref. 7). Additional background information is contained in the section, Background.

The change in shape accomplished by metalworking usually involves the creation of new surfaces and a final surface area that is larger than the initial surface area. This area increase introduces a problem when solid lubricants are used on the workpiece. In such case, a solid lubricant coating may function effectively at the temperatures and pressures involved, but may be incapable of stretching, or of flowing out, over the new surfaces created during the metalworking process. In this respect, liquid lubricants have the advantage of being able to spread over newly exposed surfaces. Solid lubricants can, however, be used effectively by applying them to the die or liner rather than to the workpiece. Solid lubricants can be utilized in several ways. They can be used as:

1. Bonded coatings on the die and liner

2. Preformed inserts at the entrace of the die

3. Pigments in greases or oil suspensions

Metalworking lubricants are usually thought of as either liquids or solids or as materials such as glasses that are applied as solids but do not lubricate until they become fluid at elevated temperatures. It is hypothesized that effective lubrication could be obtained by a lubricant acting in both the solid and liquid states. In other words, these lubricants may lubricate both below and above the melting point. The mechanism of lubrication would, of course, be different in the liquid and in the solid states. This hypothetical lubricant is not at all improbable if the candidate materials are soft, easily sheared crystalline solids that melt to form viscous liquids. An example of this type of lubricant is the lead monoxide-silicon dioxide ($PbO-SiO_2$) system; a particularly good example is the lead monoxide rich portion of the system at SiO_2 concentra-

tions lower than about $7\frac{1}{2}$ percent (ref. 26). The equilibrium phases are lead monoxide, and tetralead silicate. This material is soft in the crystalline state, melting occurs at about 1320° F, and the resulting melt is a viscous liquid that may well have capabilities as a liquid lubricant.

The objective of this paper is to explore the use of solid lubricants for high temperature metalworking, especially those solid lubricants which are effective lubricants both below and above their melting points (i.e., in both the solid and liquid states).

BACKGROUND

Although the subject of this paper is Solid Lubricants for metal working, lubrication considerations cannot be completely isolated from other problem areas in the metalworking process. An example has already been given; that is, the important problem especially with solid lubricants of supplying lubricant to the new surfaces continuously created by the plastic deformation of the work. The important problem areas in metalworking and their interaction have been summarized in excellent fashion by Backofen in reference 1.

For instance, the deformation zone or the zone just ahead of the die entrance (where the billet undergoes severe reduction in cross section) is primarily the concern of continuum mechanics. However, lubrication has its influence here because the distribution of stresses and the flow pattern of the extrusion are influenced by billet to liner and billet to die friction. Lubrication is also important to the entire process because the work required to overcome frictional resistance represents an inefficiency that is important in determining the overall force requirements of the system. Backofen (ref. 1) shows that the total work of extrusion can be expressed by the following equation:

$$W_a = W_h + W_f + W_i$$

W_a = total work of extrusion

W_h = pure deformation work for homogeneous reduction in volume from the initial to the final cross section

W_f = work done in overcoming frictional resistance at the billet-liner and billet-die interfaces

W_i = redundant or internal deformation work expended in straining not required for the shape change

The efficiency of the extrusion process is expressed as

$$\eta = \frac{W_h}{W_a}$$

and is the fraction of all work done solely for the necessary change of shape.

The work of extrusion can be expressed as the integral of the ram pressure over the distance the ram is displaced during the extrusion process. Figure 1 compares the ram pressure as a function of ram travel for an ideal (frictionless) case with a real case in which appreciable friction is experienced. The area under the lower curve is the absolute minimum deformation work required to accomplish the necessary plastic deformation. The area between the two curves represents the additional work required to overcome frictional resistance. Edgecombe (ref. 2) states that approximately 10 to 30 percent of the power requirement in steel extrusion is consumed in overcoming the friction between the billet and the container; the remainder of the power is consumed in deforming the metal. Minimizing friction with improved lubricants not only reduces power requirements, (a factor which is sometimes considered secondary because of the availability of adequate presses) but it also results in important improvements in uniformity of microstructure, surface finish, dimensional accuracy, and straightness.

It is interesting to note (fig. 1) that, as the ram progresses and the billet becomes shorter, the pressure requirement steadily decreases until it approaches the level required for deformation only. Since the frictional resistance is equal to the product of the shear strength of the sliding material and the area in shear, it is apparent that the area in shear decreases uniformly as the unextruded length of the billet becomes shorter. According to the adhesive theory of friction (ref. 27), the real area of contact between metal to metal surfaces depends only on normal load and the physical properties of the material and is independent of the apparent contact area.

Three regimes of surface contact are described by Shaw (ref. 4). In figure 2 idealized cross sections of a series of plane sliders under progressively increasing load is shown. The upper member is considered much harder than the other and the two members simulate the die-billet contact in metalworking. Real surfaces are never perfectly flat and the real area of contact occurs at the microscopic peaks or asperities. The peaks deform, first elastically and then plastically to generate a contact area sufficient to carry the load. As long as the plastic zones under the asperities act independently, that is do not overlap, (such as in Regime I where $A_r \ll A$), the real area of contact and therefore the shearing or friction force required to cause sliding, should increase directly with load and Amonton's law is obeyed. When the load is increased to the point

where A_r = A (Regime III), relative motion between the die face and the underlying material may take place by bulk subsurface flow. Since bulk shear is taking place, the force necessary is dictated only by the shear strength of the material and is independent of load; for this case, therefore, a horizontal line is drawn as the limit. For practical plastic flow situations, $A_r \neq A$ but will approach the value of A as load is increased. For such cases (Regime II, $A_r < A$), the force versus load line is intermediate between that of Regimes I and III.

It is interesting that, in a discussion of wear mechanisms, Burwell (ref. 5) shows that the wear of unlubricated metals increases linearly with applied load up to a limiting load beyond which wear increases exponentially with load (see Fig. 3). It is interesting that the change in the wear mechanism occurs as the load is increased to a level at which severe plastic deformation and subsurface shear of one or both of the sliding surfaces occurs. The serious wear that takes place during sliding under conditions of severe plastic deformation emphasizes the need of good extrusion lubricants not only to reduce pressure requirements but also to prevent metal transfer and to insure a good surface finish on the product.

LIQUID LUBRICANTS

As already mentioned, liquid lubricants have the advantage of being able to spread over the newly formed surfaces during the stretching of the work material. Liquids are, of course, commonly used as metal working lubricants. The liquids are generally oils or greases at moderate temperatures, and molten glass at elevated temperatures. One of the basic problems in using liquid lubricants for high temperature metalworking is to find lubricants with the proper viscosities over a considerable temperature range. The extrusion and forging of refractory metals such as molybdenum, require lubrication over an exceptionally large temperature range. Die temperatures may be 500° to 800° F while the billet is at temperatures in excess of 3000° F.

Turnbull, reference 6, has shown in a fundamental manner how the viscosity temperature characteristics of various classes of liquids can be approximated from thermodynamic considerations. Liquid metals, for example, have very low viscosities even at temperatures very close to melting point. Simple molecular substances and organic fluids are quite viscous near their melting points but have a rather steep viscosity temperature slope. Glasses have the flattest temperature viscosity characteristics of all known liquids and therefore have workable viscosities over a wider temperature range than other materials. This characteristic undoubtedly, partially explains the success of molten glasses in the steel extrusion process (ref. 7).

The viscosity-temperature characteristics of a common soft lime glass (from ref. 23) are shown in Fig. 4. During steel extrusion, the billet

temperature may be 2300° F initially, cooling to about 1800° F during the extrusion. The viscosity of soft lime glass at 2300° F is about 300 poises and this increases to about 10,000 poises at 1800° F. This is apparently within the range of viscosities over which molten glass will lubricate. For higher temperature processes, where soda lime glass may be too fluid, the borosilicates may be useful (Fig. 4). An interesting variety of glasses incorporates tantalum pentoxide Ta_2O_5, as the primary glass-former instead of SiO_2. The Ta_2O_5 glasses are more refractory than the silicate glasses. As shown in Fig. 4, the flow point of a 70 Ta_2O_5 - 15 K_2O - 15 SiO_2 glass is reportedly 2750° F (ref. 24). The Ta_2O_5 glasses may possibly be useful lubricants for very high temperature processes such as the extrusion of refractory alloys. The viscosity-temperature characteristics of a low-temperature glass-like material, B_2O_3 (ref. 25) are included for comparison.

Even with a choice of glasses that have considerably different softening temperatures, one of the problems in glass lubrication is still the change in viscosity with temperature. If the molten glass becomes too cool it is abrasive and can clog part of the die (see refs. 8 and 9) or cause rub-in defects or inclusions in the surface of the work. Near the maximum temperature at which lubrication is required especially with refractory metals, the viscosity of glass sometimes becomes too low and little or no lubrication is obtained (see ref. 10). A further difficulty is the removal of glass from the finished work. One of the important consideration in the selection of a glass lubricant is low chemical resistivity so that the glass can be readily removed from the work by a simple pickling procedure (ref. 11).

SOLID LUBRICANTS FOR METALWORKING

The lubricating action of solid lubricants does not depend on viscosity but rather on the shear strength of the solid. For certain solids such as the soft oxides and layer lattice compounds (e.g., MoS_2 or graphite) the shear strength is low over a large temperature range. Some solid lubricants such as MoS_2 are oxidatively unstable in air at elevated temperatures, however, there is a considerable interest in metalworking in vacuum or inert atmospheres (see refs. 12 to 14). In these atmospheres, MoS_2 is stable and it is known that it can lubricate to at least 2000° F (ref. 15) and perhaps to its melting point which is reportedly greater than 2900° F (ref. 16).

During experimental forging of molybdenum at a temperature of 2800° to 3300° F (ref. 14), an inert atmosphere of argon was maintained. Graphite, MoS_2 and various glass lubricants were compared; of these, MoS_2 was found to be the most effective lubricant. The effectiveness of graphite in terms of the amount of metal deformation per given amount of applied force was about the same as that of MoS_2. The forgings produced using MoS_2 had a better surface, however. The surfaces obtained using glass lubricants were not as good as those obtained with MoS_2 or with no coating at all when the metal was allowed to oxidize to a limited extent and some lubrication was obtained from the molybdic oxide (MoO_3) so formed.

It has been reported (ref. 17) that a number of solid lubricants are useful in the extrusion of 4340 steel at an extrusion temperature of 2100° F and for the extrusion of molybdenum at 3400° F. The effective lubricants included flake graphite, usually mixed with a metal oxide (such as tin oxide, copper oxide, manganese oxide, or lead oxide); sodium silicate and graphite also had useful properties. Mixtures of molybdenum disulfide and sodium silicate as well as tungsten disulfide and sodium silicate were also evaluated; of these two, tungsten disulfide plus sodium silicate was more effective. Other useful solid lubricants included a proprietary phosphate composition, Phosphotherm RN. Boron nitride showed no encouraging results regardless of what binder or carrier system was used. Promising preliminary results were obtained with some salt systems in preliminary screening tests but they were not evaluated in the actual extrusion process.

A number of plastics were also evaluated. Plastics, of course, decompose at the extrusion temperature but the ablation of these materials requires time and the extrusion was completed before total decomposition took place. Promising results were obtained with a mixed epoxy and flake graphite and with the Dupont polyimide, SP-1. Even better results were obtained with the graphite filled polyimide, SP-2. These plastics plus Bakelite and also graphite can be effectively used as preforms that are inserted into the entrance portion of the extrusion die as shown in Fig. 4. This technique improves die life considerably and places the material at the points of maximum stress. According to the authors of reference 17, die life is further improved by coating the entrance surface of the die with zirconium oxide. The zirconium oxide-coated die, as well as the plastic preform assembly, is shown in Fig. 4. 5

Soft metallic coatings have also been used as lubricants in metal-working processes. In reference 18 a description is given of how copper was effectively used as a metallic lubricant on tantalum. Copper was electroplated on tantalum which could then be drawn down to a wire of 0.0004-inch diameter. After drawing, the copper was removed with nitric acid. In another study (ref. 19), aluminum alloys were clad with pure aluminum. This helped reduce the die friction but pure aluminum cold-welded to the tool and eventually caused clogging of the die. Bailey and Singer (ref. 20) studied the lubricating properties of cadmium oxide, molybdenum disulfide, graphite, and graphite cadmium oxide mixtures at temperatures to 600° C and metal deformations to 90 percent. They reported that friction with cadmium oxide was low to about 200° C and increased between 200° and 300° C. The friction with MoS_2 slowly increased with temperature and reached a maximum at 500° C. This undoubtedly corresponds to the formation of molybdic oxide, MoO_3, by reaction of MoS_2 with oxygen in the air. Graphite was good to 300° C where a peak in the friction coefficient occurred. The friction coefficient decreased again and was good at about 500° C. The addition of cadmium oxide to graphite eliminated the high friction peak at 300° C.

These results, especially with the graphite-cadmium oxide mixtures are in remarkable agreement with the lubricating properties of the same materials under the more moderate conditions of elastically loaded slider specimens. For example, in reference 21 it is demonstrated that graphite lubricates from room temperature to 200° F but exhibits high friction coefficients from about 300° to 800° F and then lubricates again at 1000° F. When cadmium oxide is added to the graphite, a low friction coefficient was obtained over the entire temperature range.

NASA RESEARCH WITH SOLID LUBRICANTS

The above examples indicate that solids can be used as successful lubricants for metalworking. They should especially be considered for high temperature metalworking where lubrication with organic fluids may not be feasible and especially where the viscosity temperature characteristics of available inorganic liquids such as molten glasses may not be satisfactory. Other lubricants have been studied at NASA for some time. Some formulations have been developed that show promise for lubrication at elevated temperatures. These compositions were primarily intended for the lubrication of elastically loaded bearings and for temperatures up to about 2000° F. However, it might be helpful to review the lubrication properties of some of these materials and perhaps to speculate how they might be of use in metalworking. The lead monoxide silicon dioxide ($PbO-SiO_2$) composition has already been mentioned. The friction temperature characteristics of a bonded lead monoxide coating is shown in Fig. 5. Low friction was obtained under conditions that produced high surface temperatures, that is, either high ambient temperature or high sliding velocity; the best friction results were, therefore, obtained under the most severe conditions. This coating was not evaluated at temperatures above 1250° F because the $PbO-SiO_2$ eutectic melts at 1370° F and it was not desired to melt the coating. However, the hypothesis discussed in the introductory portion of this paper (that some solid lubricants might lubricate both below and above their melting points) led us to check this hypothesis experimentally with the same $PbO-SiO_2$ composition as in Fig. 6. The substrate material in this case was a nickel-base alloy, Inconel. The sliding velocity was 6 feet per minute. The results are shown in Fig. 7. The results show reasonably low friction coefficients at temperatures below and above the melting point of the $PbO-SiO_2$ eutectic ($PbO + 4PbO \cdot SiO_2$). Friction coefficients were also reasonably low even at temperatures above the melting point of PbO. There are discontinuities at temperature near both melting points.

The results appear to confirm the concept that some compositions may be capable of lubricating in both the solid and the liquid state, thus providing an extended temperature range for lubrication. This technique may not be effective for bearing lubrication because the molten oxides are extremely corrosive to metals during long duration exposure; the technique may, however, be useful for certain metalworking applications such as extrusion.

The results of friction studies reported in reference 15 indicate that some other materials may lubricate in both the solid and the liquid state. Solid bismuth oxide, (Bi_2O_3) for example, is a fair solid lubricant from room temperature to the melting point of the oxide at 1540° F, Fig. 8. The friction coefficient was about 0.18 just below the melting point then dropped abruptly to 0.08 as the compound melted. The friction coefficient was below 0.2 up to the maximum temperature studied, 1800° F. Results obtained with boric oxide (B_2O_3) and molybdic oxide (MoO_3) are also shown in Fig. 8; these two compounds had little or no lubricating ability until they melted. As a caution it should be noted that the molten oxides are not only quite corrosive to metals but that a sharp friction peak sometimes occurs just beyond the melting point; friction drops rapidly as the temperature is further increased.

Ceramic compositions have been formulated to lubricate nickel base alloys to 1500° F. The friction and wear properties of some of these are shown in Fig. 9. The important characteristic of these coatings is that they are not hard or brittle at room temperature, as most ceramics or enamels, but are soft enough to be scratched readily with unhardened steel. The wear and friction coefficient of Inconel specimens coated with these compositions were considerably lower than with the uncoated specimens. Further reduction in friction and wear was obtained by using calcium fluoride (CaF_2) with one of the ceramic formulations as a binder. The friction and wear of ceramic bonded CaF_2 on Inconel-X is shown in Fig. 10. Subsequent work (ref. 22) has indicated that ceramic-bonded CaF_2, which melts at 2480° F, can lubricate to at least 2000° F under certain conditions. Some results (ref. 22) with ceramic-bonded CaF_2 at temperatures to 1900° F are shown in Fig. 11.

CONCLUSIONS

In this paper, an attempt has been made to review some of the uses of solid lubricants in metalworking. Background information and some discussion of liquid lubricants has been included to establish the proper perspective and to determine where solid lubricants fit into the overall picture. The major conclusions drawn from this study are:

1. Solid lubricants can be useful in metalworking especially in applications such as the extrusion of refractory alloys where lubrication must be obtained over a very wide temperature range. It may be possible to extend the temperature range of usefulness for some solid lubricant by using them beyond their melting points. Certain oxides in particular (such as lead monoxide and many others) are soft and nonabrasive when in the solid state and form viscous fluids with lubricating capabilities when molten.

2. Many solid lubricants are oxidatively unstable in air at elevated temperatures. This difficulty can sometimes be alleviated, however, by performing the metalworking operation in an inert atmosphere. An inert atmosphere is often desirable, especially in the working of refractory alloys in order to minimize oxidization. It is well known that the refractory metals have high strength but poor oxidation resistance at elevated temperatures. The same atmosphere that protects the metal, will therefore serve to protect the lubricant.

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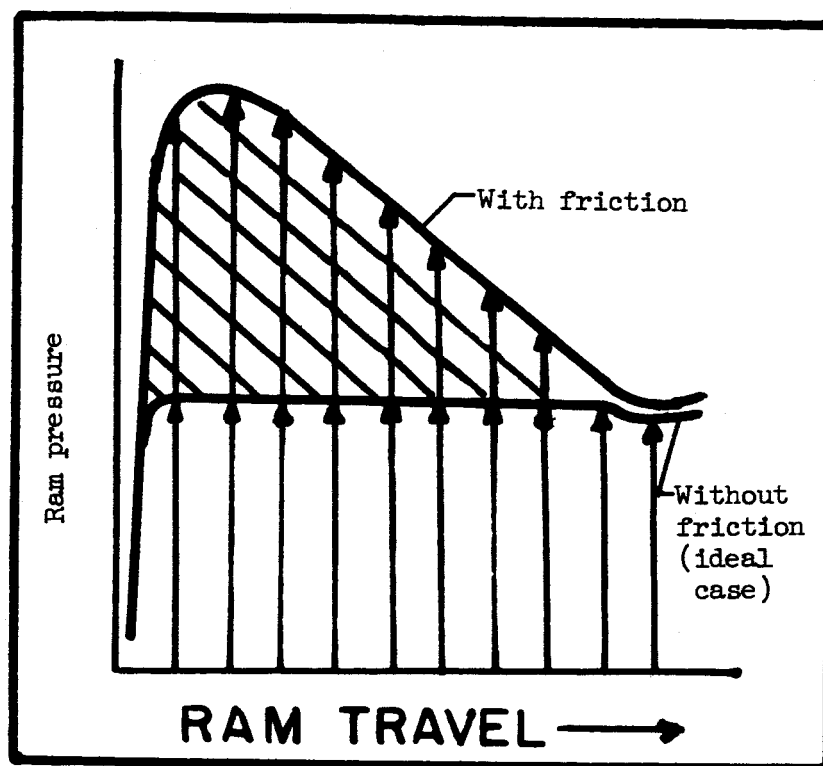
With friction

Without
friction
(ideal
case)

Ram pressure

RAM TRAVEL ➔

Figure 1. - Effect of sliding at billet-container interface during extrusion (after Backofen, ref. 1).
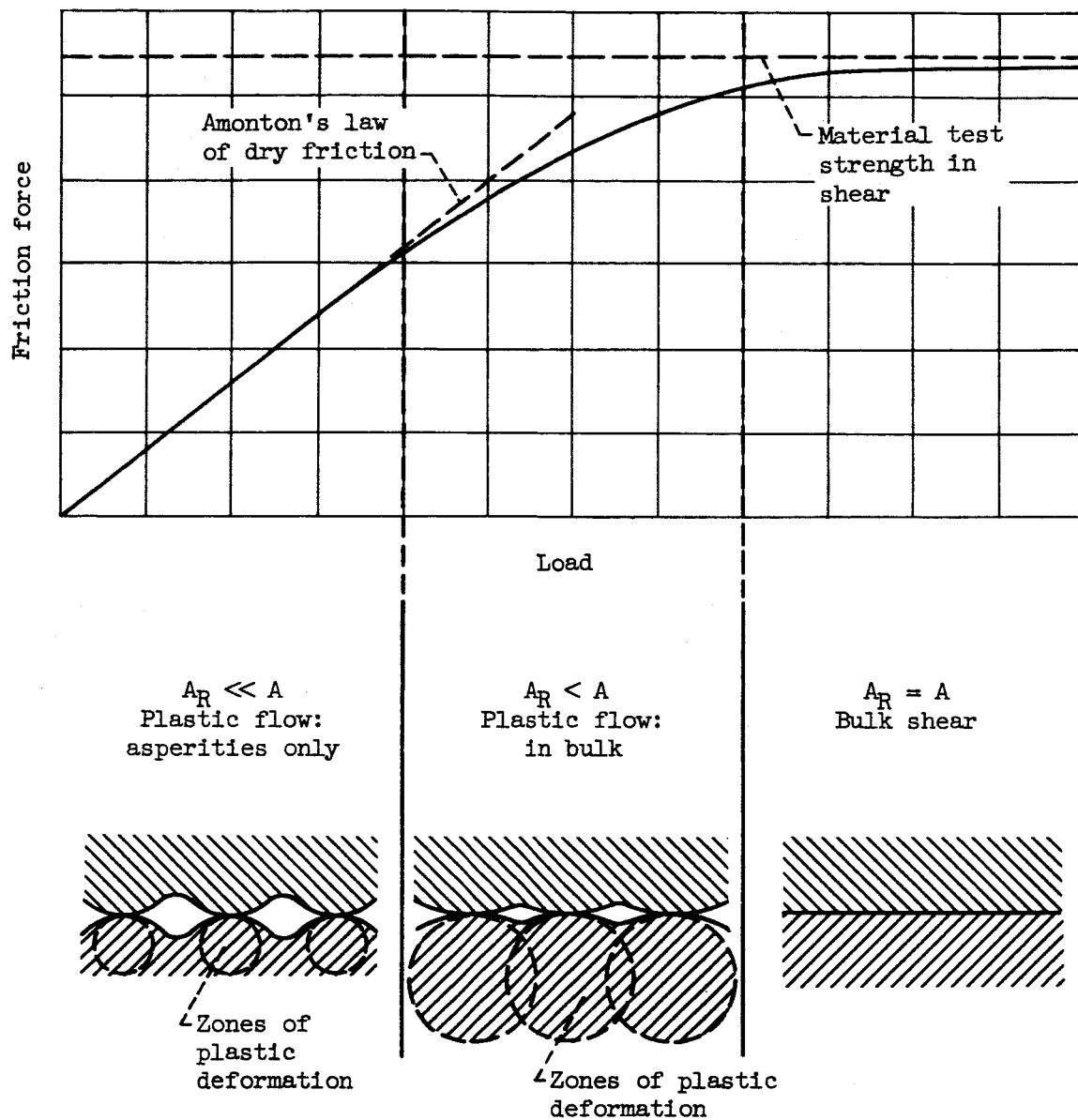
Figure 2. - The influence of plastic deformation on friction (After Shaw, ref. 4).
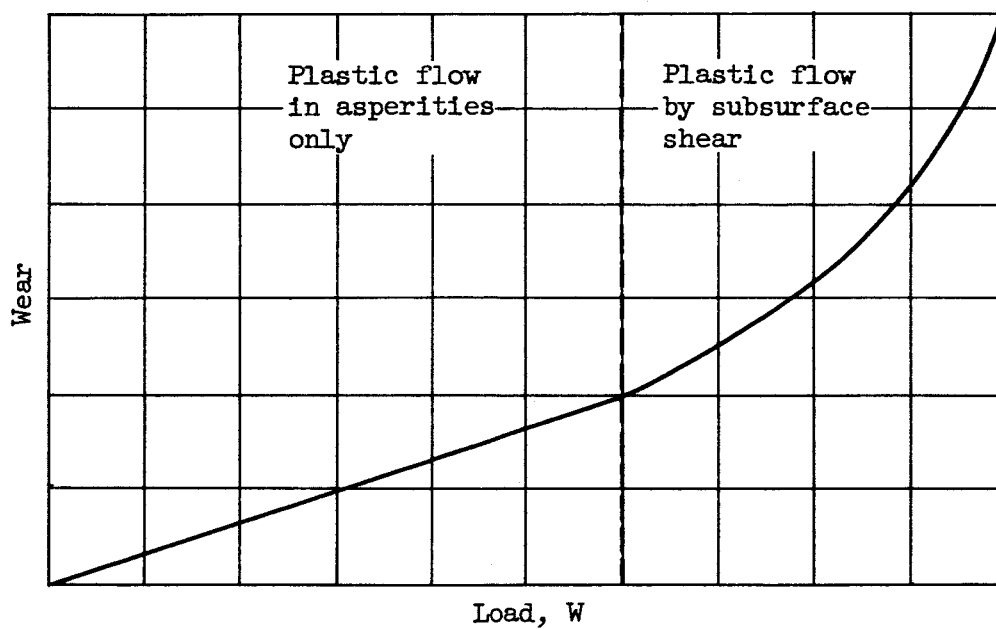
Figure 3. - The influence of load on the wear of specimens in sliding contact (after Burwell, ref. 5).
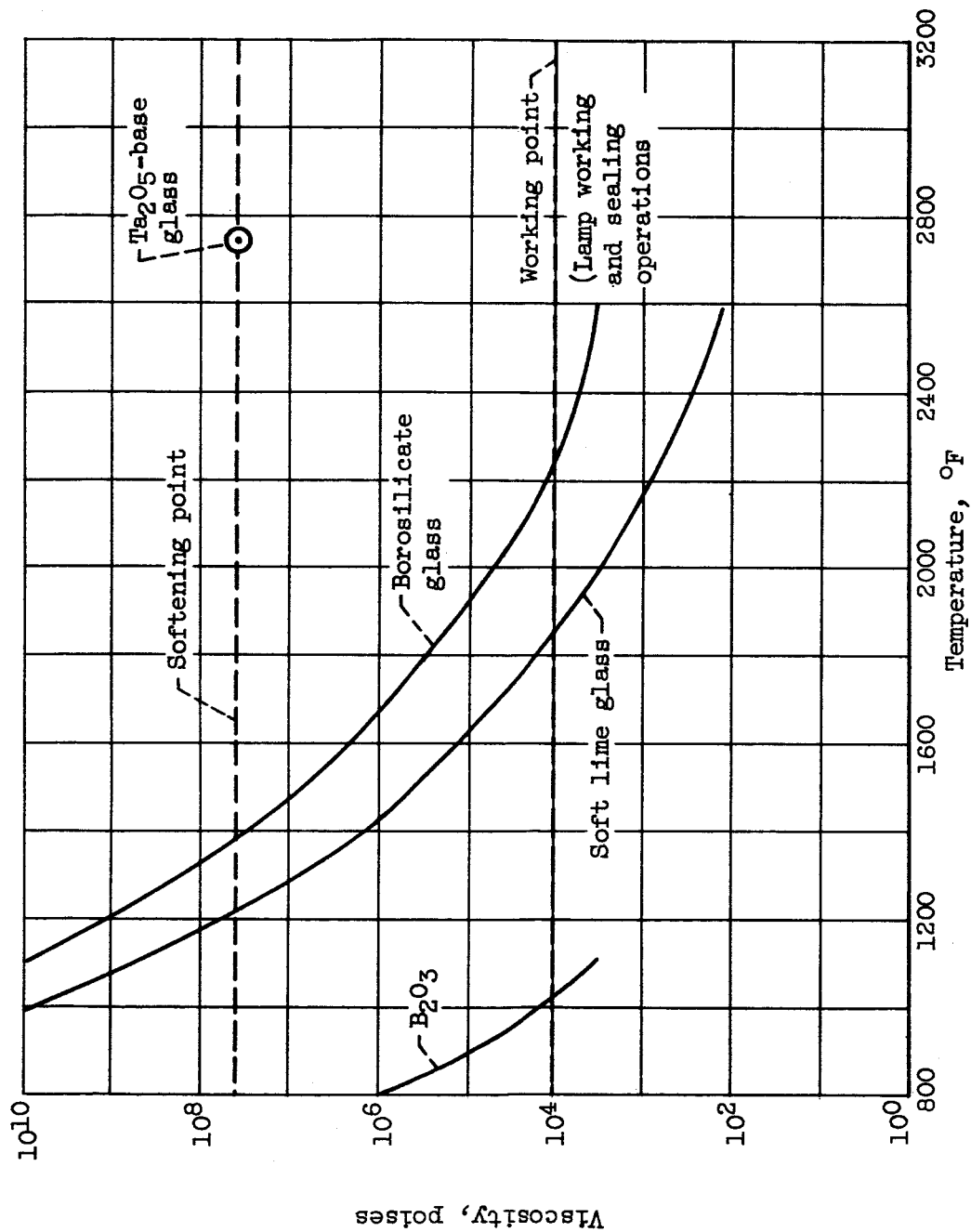
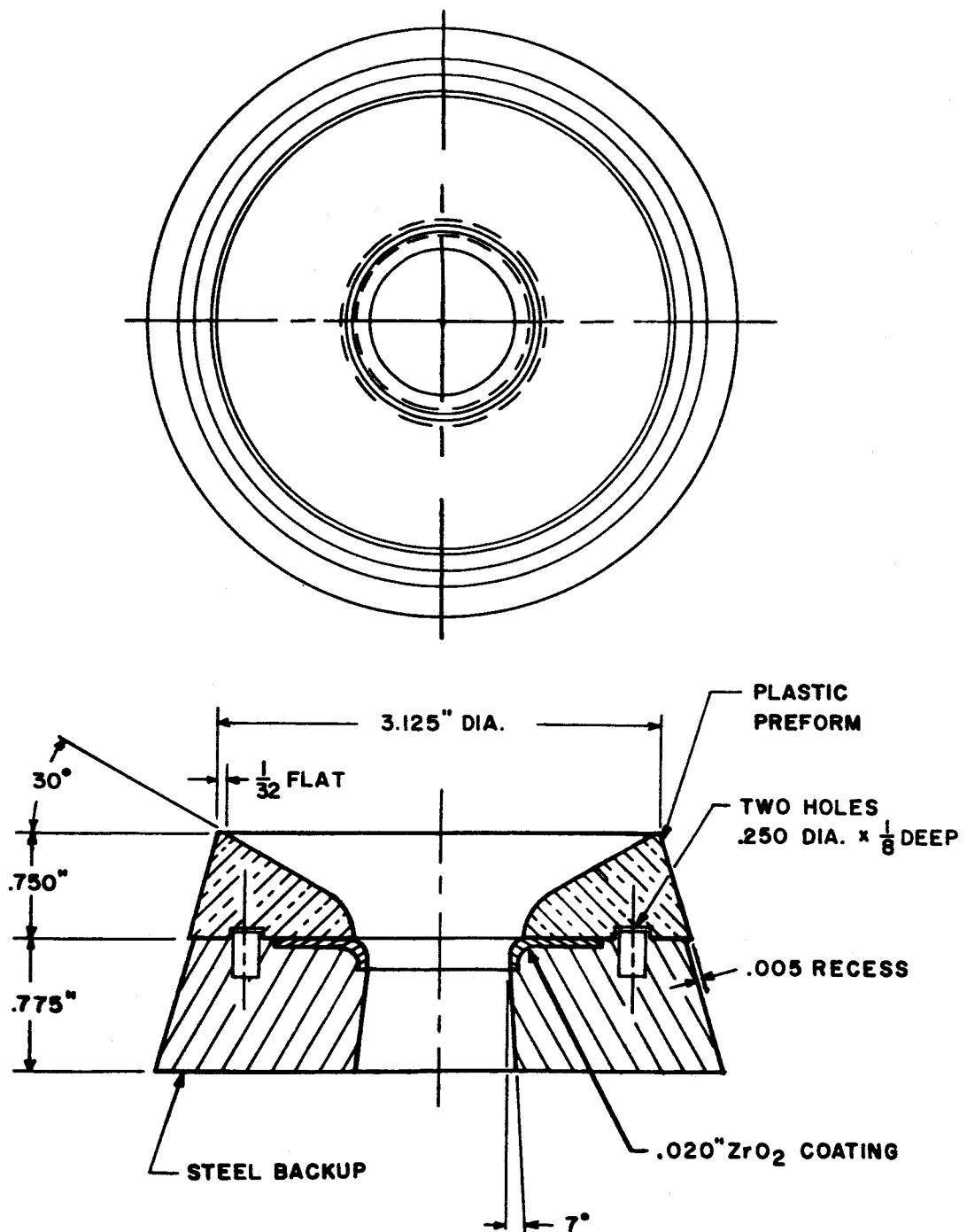Figure 4. - Viscosity temperature characteristics for glasses and glass-like materials.

Figure 5. - Assembly sketch of two piece plastic-tool steel extrusion die. (ref. 17).
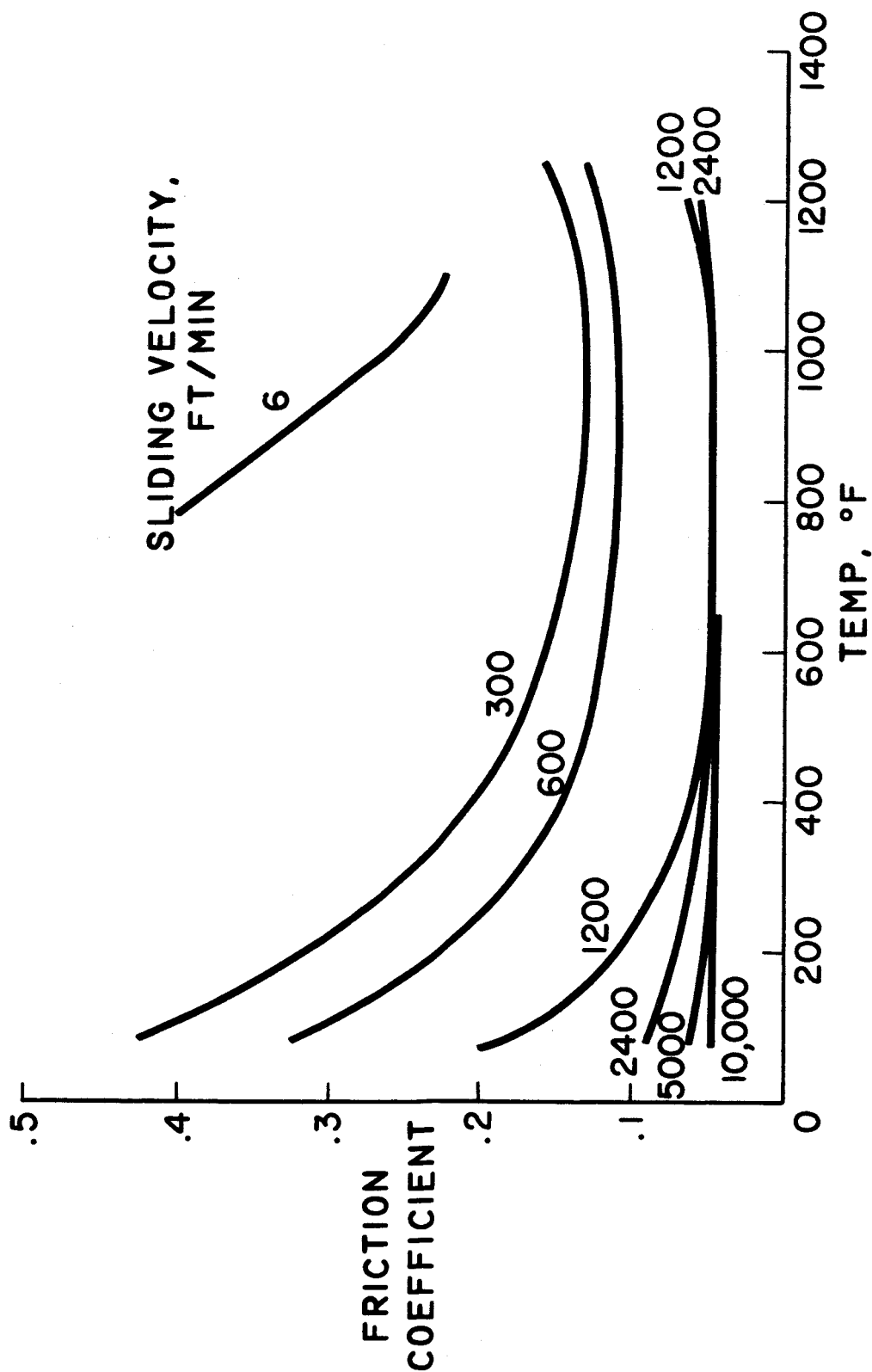
Figure 6. - Effect of sliding velocity and temperature on friction properties of PbO-SiO$_2$ coatings bonded to 440-C stainless steel. Coating thickness, 0.001 in.; load, 1000 g.
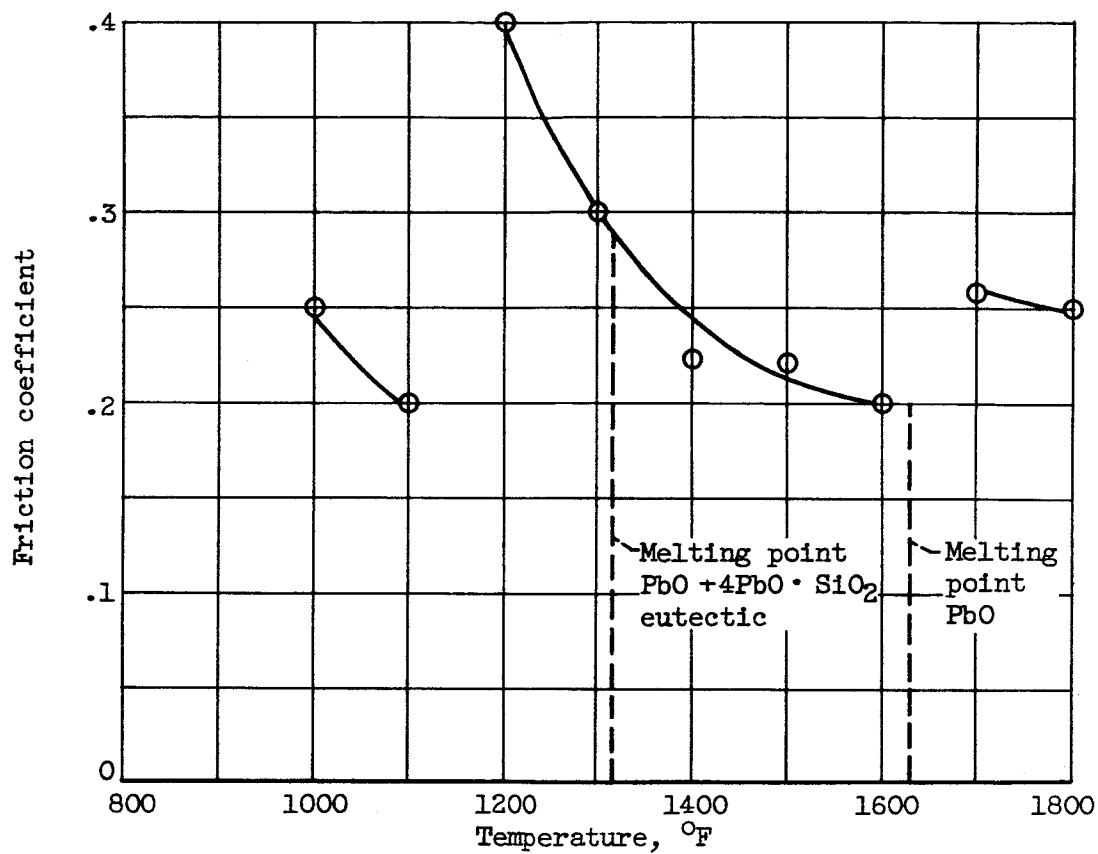
Figure 7. - Friction-temperature characteristics of
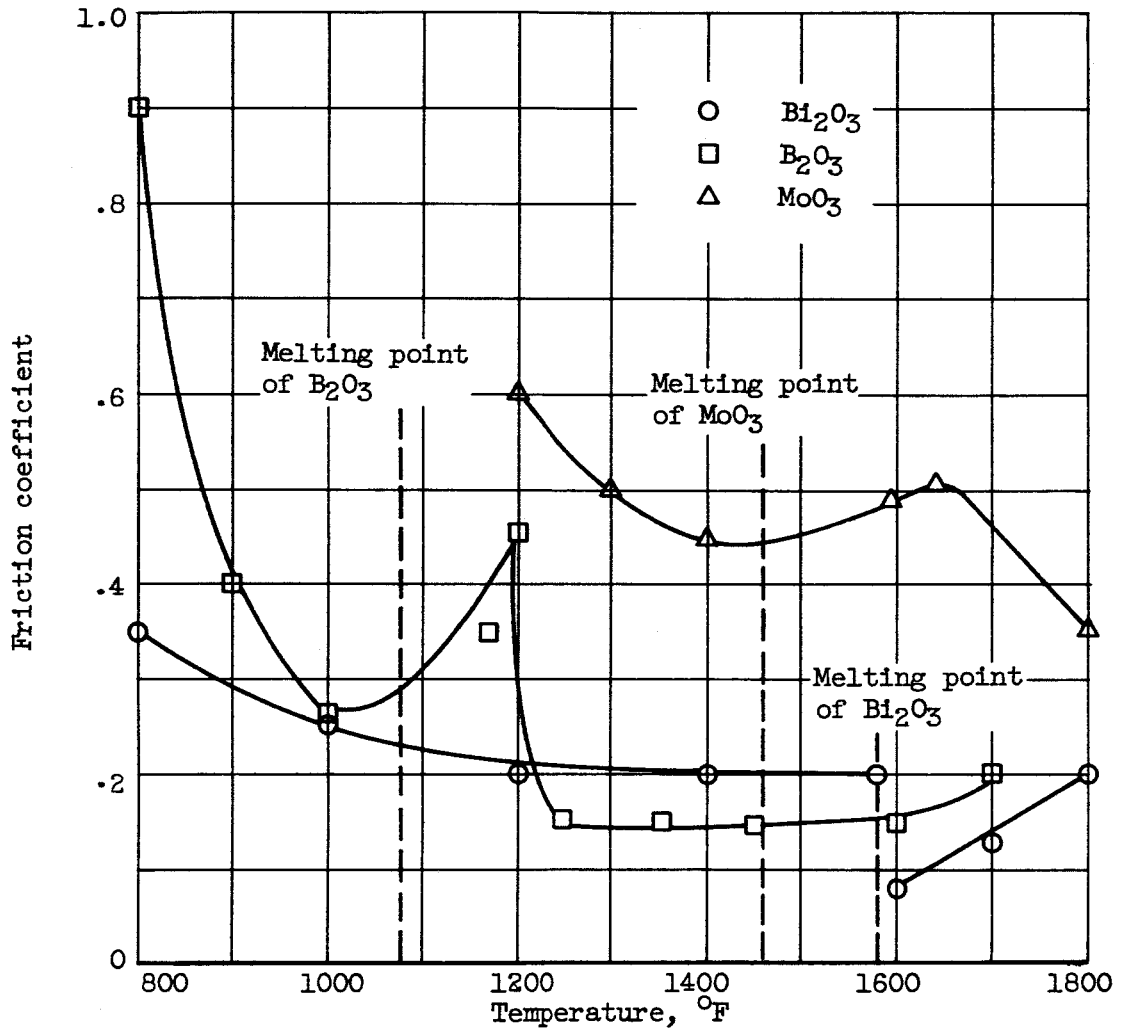95PbO-5SiO$_2$ on inconel at low sliding velocity (6 ft/min).

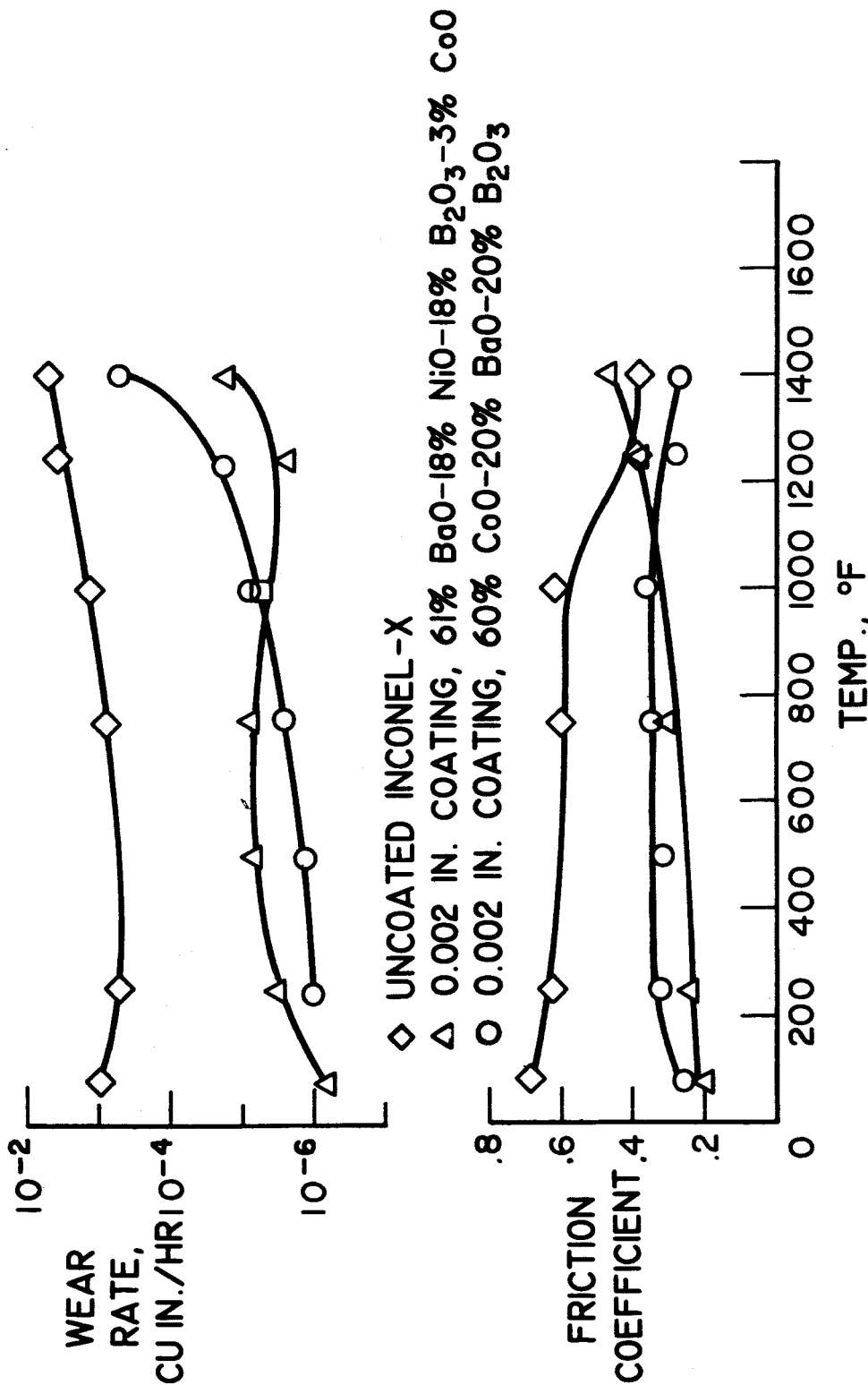Figure 8. - Friction-temperature characteristics of several oxides below and above their melting points.

E-2759

Figure 9. - Effect of temperature on lubricating properties of a CoO-base and a BaO-base coating on Inconel-X.
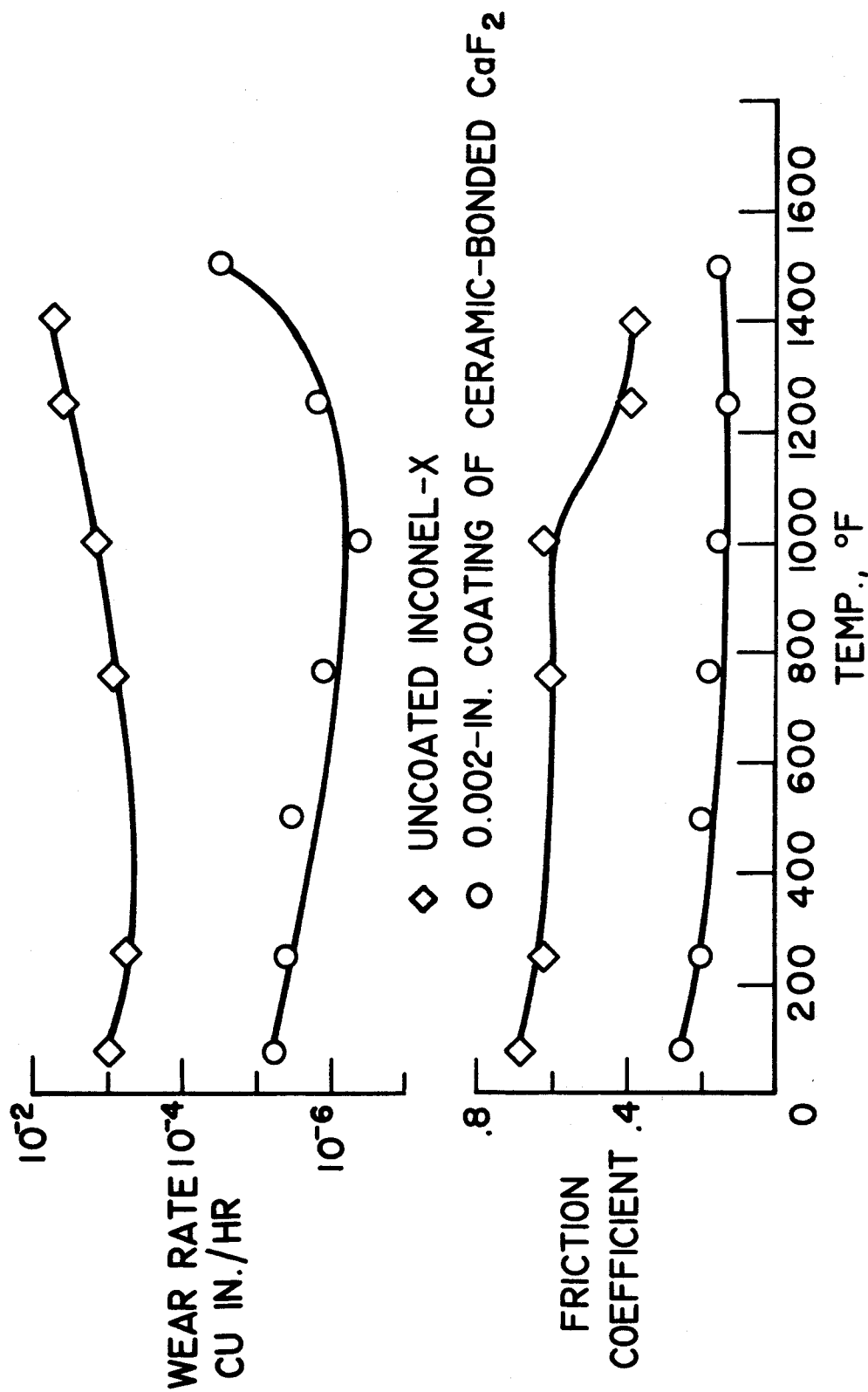
UNCOATED INCONEL-X

0.002-IN. COATING OF CERAMIC-BONDED CaF$_2$

WEAR RATE CU IN./HR

FRICTION COEFFICIENT

TEMP., °F

Figure 10. - Effect of temperature on lubricating properties of ceramic-bonded calcium fluoride coatings on Inconel-X.

Rider wear rate, cu in./hr (log scale)

10,000×10⁻⁶

1,000

100

10

1

.1

Friction coefficient

.4

.2

0

Rene 41 on unlubri-
cated Rene 41

Cast Inconel on unlu-
bricated Inconel X

Rene 41 on CaF₂-
lubricated Rene 41

Cast Inconel on CaF₂-
lubricated Inconel X
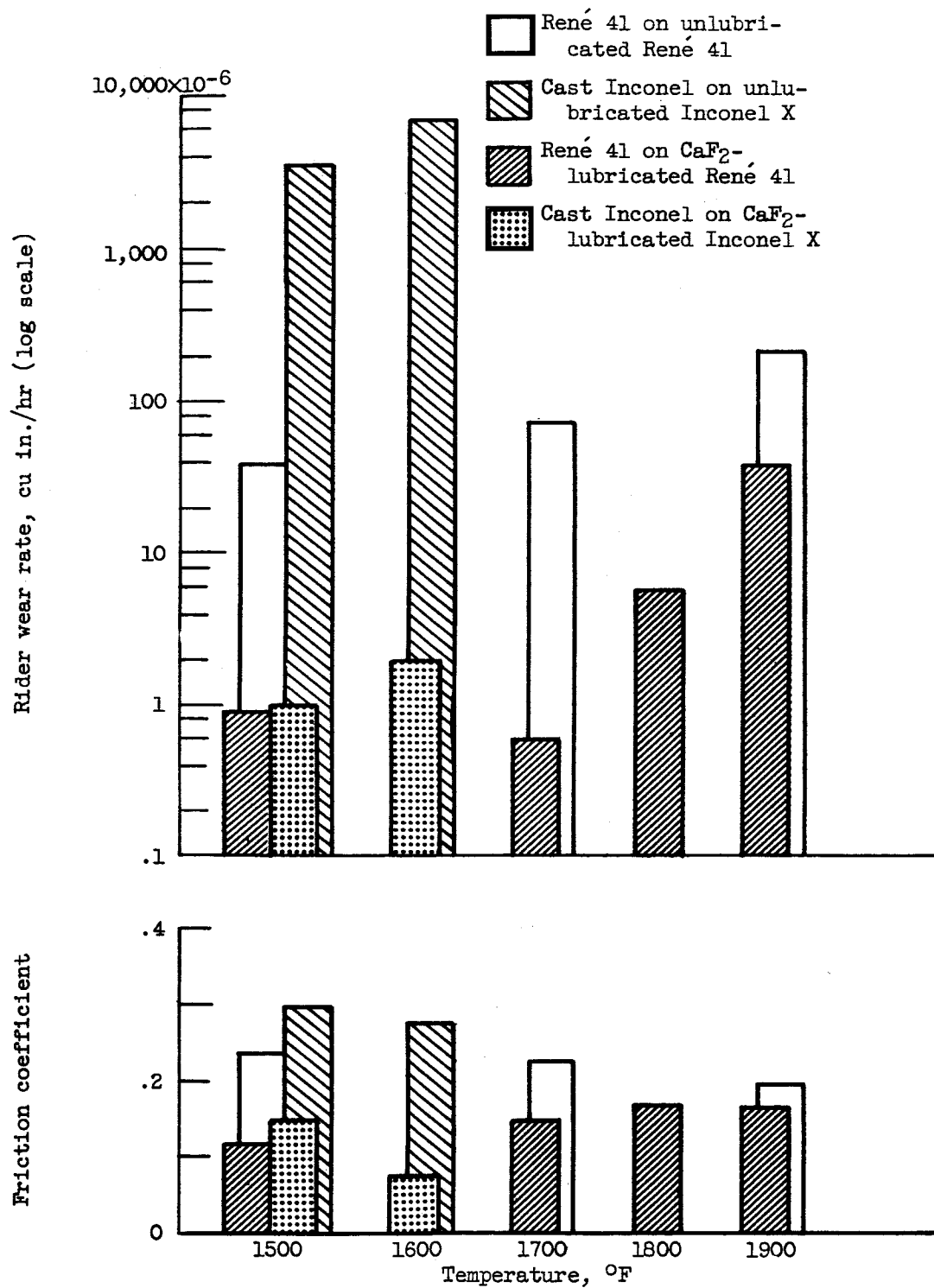
1500   1600   1700   1800   1900

Temperature, °F

Figure 11. - High-temperature friction and wear
properties of CaF₂-lubricated and unlubricated
metals.